PROCESS FOR INSPECTING MONOCRYSTALLINE MATERIAL FOR PRECIPITATION OF IMPURITIES

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a process for the inspection of monocrystalline material for the presence of precipitations of impurities, whereby discrete crystal regions are irradiated with wave or particle rays, the intensity distribution of rays diffracted under Bragg conditions is recorded, and a determination of the static Debye-Waller factor is made by fitting the recorded intensity values to a specified theoretical function.

2. Background of the Invention

Since the control and optimizing of the manufacture and processing of single crystals play an important role in many industrial applications, great significance is attached to processes which make it possible for the manufactured crystals to be inspected and the deviations from the ideal crystal structure detected. A typical use of such processes is the inspection of Silicon (Si) wafers which are to be reprocessed for use in the semiconductor and microelectronics industries.

Si wafers are generally in the form of monocrystalline discs with typical dimensions of approximately 0.4 mm thick and 200 mm in diameter. The wafers obtained, after the crystal drawing, contain an oxygen component on the order of magnitude of 10 ppm, with the exception of the active surface layer, approximately 20 mm thick, which is practically oxygen-free. During reprocessing the wafers are heated during tempering, whereby at approximately 700° C., the previously atomic oxygen tends to form SiO_2 molecules. The SiO_2 molecules can substitute for Si atoms in the crystal lattice, which causes local distortions of the lattice structures.

It has been shown that the distortion of the lattice has an attractive effect on additional SiO_2 molecules, which accumulate in the plane structure. In this manner, thin platelets of SiO_2 precipitations form, which causes stresses in the crystal. These precipitations also have the characteristic of gettering sinks. Gettering is the process by which existing defects are annihilated and impurities are removed. More specifically, intrinsic gettering occurs in Si crystals when the precipitations attract foreign atoms internally; in particular metal atoms such as Ag, Cu, etc., which originate from impurities in chemicals required during the manufacturing process for semiconductor elements, e.g. etching fluids. As a result of the intrinsic gettering, the yield during the manufacture of micro-electronic semiconductor components is significantly increased. A prerequisite of intrinsic gettering is that the intrinsic gettering layer is underneath the subsequently electrically active surface layer of the wafer. The gettering layer helps purify the electrically active surface layer.

The stresses caused by precipitations in the crystal lattice are reduced at the elevated temperatures of more than 1000° C., which elevated temperatures occur during the subsequent process stages, and result in stacking errors. The stacking errors cause extensive distortion fields, which extend into the oxygen-free surface layer. It is desirable, to "suck out" impurities from the surface layer and to fix the stacking errors in the lower crystal layers. Again, the intent is to purify the electrically active surface layer. It is therefore desirable to design

the treatment processes for the wafer so that there is a uniform distribution and homogenization of the SiO_2 precipitations, to guarantee the most uniformly possible intrinsic gettering and thus to prevent disruptions of the electrically active surface layer.

Accordingly, there has long been a demand for a process by way of which the impurity precipitations can be made observable and measurable, in terms of density, size, and distribution, in the wafer. This is true in particular for a process which can be performed repeatedly in individual process steps or even in situ, i.e. during tempering of the wafer in a furnace.

The Japanese publication "Japanese Journal of Applied Physics, Volume 27, No. 8, June 1988, Pages 1081 to 1087 (S. Iida et al: Measurement and Analysis of the Static Debye-Waller Factor of Cz-Silicon with Small Oxygen Precipitates) discloses a method of the type described above, which is suitable for laboratory-scale tests on Si specimens for SiO_2 precipitations both of large extent and low density, and also for such microprecipitations whose dimensions are small in relation to the resolution of the X-ray diffraction intensity distribution recorded. The intensity distribution is thereby recorded photographically, and the blackening of the negative can be evaluated, for example, with a microdensitometer. For this purpose, specimens and sharply collimated beams must be oriented in a fixed manner, and the specimens and the negative to be exposed must be at the smallest possible distance from one another.

OBJECT OF THE INVENTION

Accordingly, the object of this invention is to create a process for such inspections which, as indicated above, can be used universally during the various steps of a manufacturing process, allows the use of different and large specimen thicknesses, makes possible a high resolution, is flexible with regard to changes in the wavelength of the radiation used, and which, in particular, meets the requirements for automation.

SUMMARY OF THE INVENTION

In the process according to the invention, the specimen to be tested is typically a crystal disc and is irradiated in Laue transillumination geometry. Thus, Bragg reflections occur on lattice planes of the lattice, which lattice planes are perpendicular to the crystal surface being irradiated. The orientation of the lattice planes of the crystal must be known or determined. Patents which involve the determination of crystal lattice plane orientation are U.S. Pat. No. 4,788,702 entitled "Orientation of Crystals" and U.S. Pat. No. 4,217,493 entitled "Hemispherical Laue Camera." The diffracted beam arrives at a photon detector, and the absolute reflection factor R can be determined in the manner of the prior art. The absolute reflection factor R is essentially the fraction of irradiation which is "reflected," a comparison of the incident intensity to the reflected intensity. Then the orientation of the crystal disc is varied, by rotating it around a first axis which is perpendicular to the diffraction plane defined by the incident and the diffracted beam. The reflection factor, as a function of the angle of rotation θ around the first axis, is recorded as the stored signals and leads to the Bragg reflection peak. An integration over the angle of rotation θ gives the integral reflection factor

$$R_{int} = \Sigma R_i \times d\theta$$